PHYSICAL PROPERTIES OF RARE EARTH-COBALT MAGNET MATERIALS

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Army Materials and Mechanics Research Center

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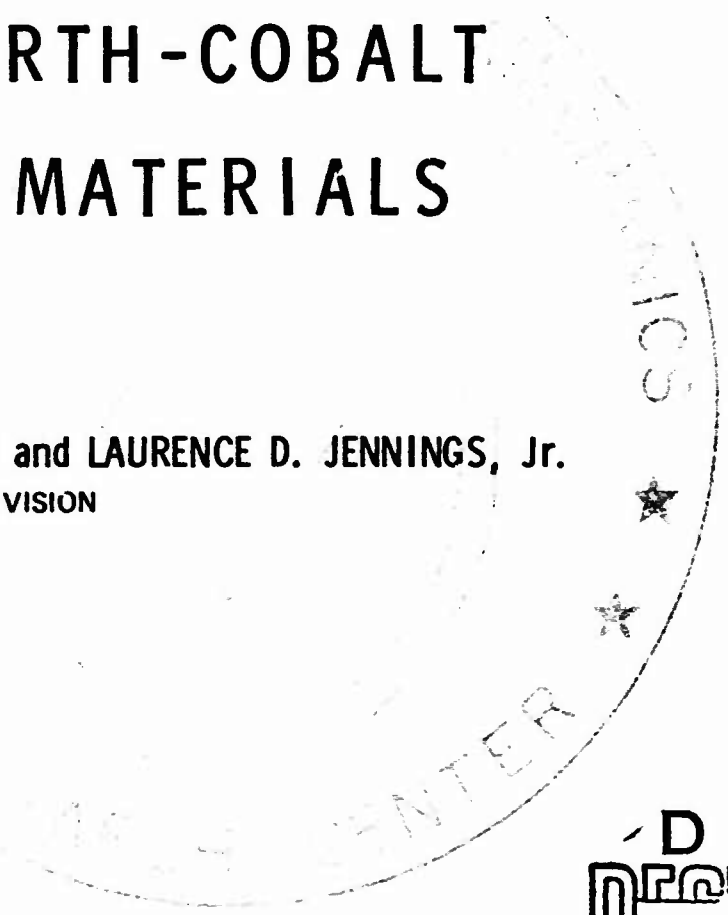

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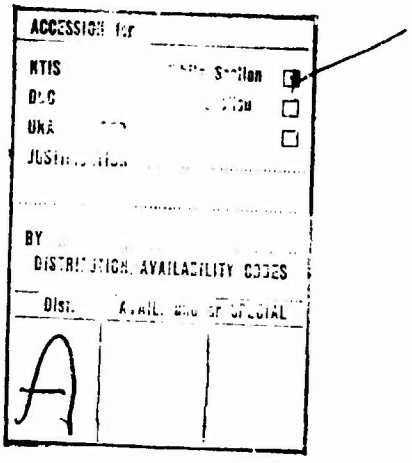

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13. ABSTRACT

Studies have been undertaken to define the changes in properties which occur in rare earth-cobalt magnet materials in the temperature range up to 1000 C. A wide range of types of behavior of the time dependence of the magnetization at relatively low temperatures is observed. It is shown that the simple mechanisms for this time dependence do not apply. Unusual types of irreversibility are displayed, and these may be useful in applications, e.g., in some cases it would be advantageous to remagnetize after a number of hours at the operating temperature. X-ray measurements were carried out at high temperatures. Although some lattice parameter values were determined, it was not possible to gain much structural information because of rapid sample deterioration. Details of the mechanism of this deterioration are reported. (Authors)

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Technical Report by
DAVID R. CHIPMAN and LAURENCE D. JENNINGS, Jr.

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ABSTRACT

Studies have been undertaken to define the changes in properties which occur in rare earth-cobalt magnet materials in the temperature range up to 1000 C. A wide range of types of behavior of the time dependence of the magnetization at relatively low temperatures is observed. It is shown that the simple mechanisms for this time dependence do not apply. Unusual types of irreversibility are displayed, and these may be useful in applications, e.g., in some cases it would be advantageous to remagnetize after a number of hours at the operating temperature. X-ray measurements were carried out at high temperatures. Although some lattice parameter values were determined, it was not possible to gain much structural information because of rapid sample deterioration. Details of the mechanism of this deterioration are reported.

FOREWORD

This report covers work done in the period Jan. 1 to June 30, 1972, under the general title *Physical Properties of Rare Earth-Cobalt Magnet Materials*. The work is sponsored by the Advanced Research Projects Agency under ARPA Order No. 1914, Program Code No. 2D10. The work was carried out at the Army Materials and Mechanics Research Center, Watertown, Massachusetts, 02172, by the principal irvestigators, D. R. Chipman and L. D. Jennings (Phone: 617 - 926-1900, Ext. 386 or 375).

INTRODUCTION

In our first progress report,[1] we considered the various physical properties of rare earth-cobalt (RE-C) magnet materials from the point of view of applications. In view of the result that stresses of practicable value have negligible effect on the properties of RE-C magnets, it was concluded that the most important outstanding problem was the mechanism for degradation of magnetization, especially at elevated temperatures. This degradation is not only important from the point of view of applications, its characterization also delimits the possible mechanisms of magnetization reversal. As this mechanism becomes better understood, it will be possible to manufacture more powerful and/or less expensive magnets.

It is now well established that the magnetic hardening of $SmCo_5$-based magnets is drastically affected by heat treatment in the range 750 C to 1000 C.[2,3] We have therefore carried out two types of studies to clarify the situation outlined above. Firstly, we have made additional measurements of the time dependence of magnetization under various conditions. Secondly, we have initiated x-ray studies in the temperature range above 750 C. There are several interesting effects which can be revealed by such measurements. It may be that the magnetic hardening is brought on by a phase change in this temperature range. Even if such a change is not found, the results would give information about the stresses introduced as the magnet is cooled through the Curie temperature and also about the magnetostriction which is interesting from a fundamental point of view. Unfortunately, these measurements present difficulties which are discussed in detail below and it may not be possible to do all the desirable work.

TIME DEPENDENCE OF MAGNETIZATION

The decay of magnetization under various conditions gives some definition of the possible mechanisms of magnetization reversal. It is also important from the point of view of applications. We have therefore extended the previous work[1] to additional cases, primarily to samples given alternate heat treatments. The hope is that through this procedure there will not be any change in those variables that are difficult to control precisely, such as oxide content, particle size distribution, or alignment. A number of cases were studied, but it is not possible at this time to make generalizations which allow an easy visualization of the situation. Instead we give a few typical results to illustrate possible types of behavior which have been observed. More such studies are being carried out to more fully characterize the situation.

Experimental

It was necessary to modify the previous apparatus somewhat in order to carry out time-dependent studies effectively. Additional instrumentation was incorporated so that the time dependence was recorded automatically and the integration circuits were improved in order to obtain greater

stability. The temperature control circuits were improved so that it was possible to start meaningful measurements about 10 seconds after the sample reached the control temperature and this temperature was held stable to better than 0.1 C. Because measurements under strained conditions were no longer required, it would have been possible to redesign the sample chamber and pickup coils to allow a smaller magnet gap and thence higher magnetic fields. On the other hand, a large gap minimizes interactions between the sample and the magnet so that, for example, the demagnetizing field on a sample is not changed appreciably when it is inserted in the magnetometer. Therefore the magnetometer-magnet arrangement was left essentially unchanged.

Some preliminary heat treatments were carried out in an argon atmosphere in sealed silica ampoules. Even though the samples were wrapped in molybdenum foil, the sample surfaces showed significant deterioration after this treatment, as discussed in more detail later. All heat treatments of actual magnets were made with the sample held in a closely fitting container of $SmCo_5$. There was very little chemical deterioration of a sample under these conditions.

Results

From the previous work[1] it had been shown that magnets from lot A showed a distinctive and appreciable time dependence on their initial run at 215 C (see Figure 1a). Because later behavior has been so different from this initial behavior, we report mostly results on samples from this lot.

Common treatments of magnetic time effects[4] invoke concepts such as fluctuating effective magnetic fields or diffusion of materials which lock domain walls. Under certain simplifying assumptions, these pictures yield a logarithmic time dependence, which is often observed in practice.[5] The behavior of $SmCo_5$ magnets is more complicated.[1] Typically a virgin run at 215 C showed an exceptionally large time dependence and also an appreciable permanent loss, that is, an unrecoverable loss in the open circuit magnetization. Although it was not possible to quantitatively relate the time dependence to the permanent loss, it is tempting to speculate that they are related. For example, it might be that the initial large time dependence and the permanent losses are brought on by diffusion and that the more moderate time dependences noted in later runs are brought on by fluctuating effective fields.

To test the applicability of these ideas, sample A2 was saturated in a 9.0 kilo-oersted applied field and heated 215 C. Under these conditions the demagnetizing field is about 3.5 kilo-oersteds. Therefore the internal magnetizing field in this experiment was about 3/2 as much as the demagnetizing field in the previous open circuit experiments, and the sample was observed to be effectively saturated. Two successive "heat treatments" of 200 minutes each were carried out on this sample. Reference to Figure 1a shows that this time was considerably more than that required to effect the marked drop noted earlier. If a simple diffusion process were involved,

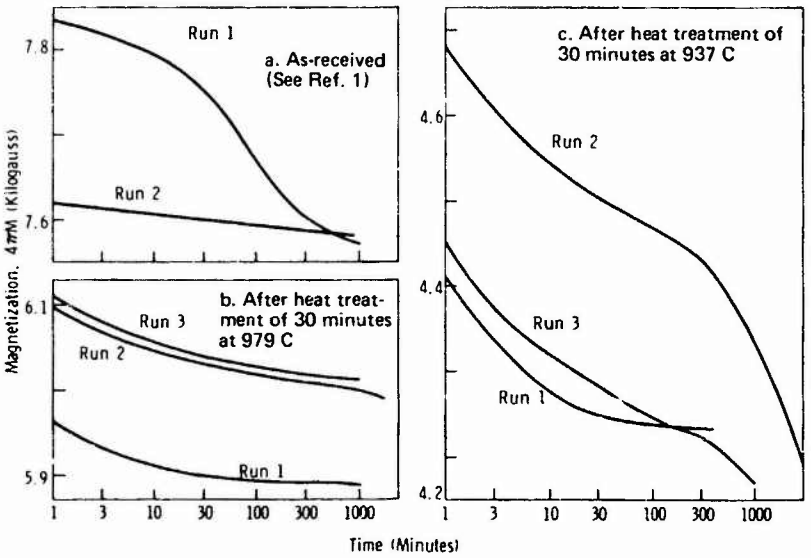

Figure 1. Open Circuit Time Dependence of the Magnetization for Sample A at 215 C. To Obtain the Irreversible Loss, Add 9.2% and Compare to Figure 2.

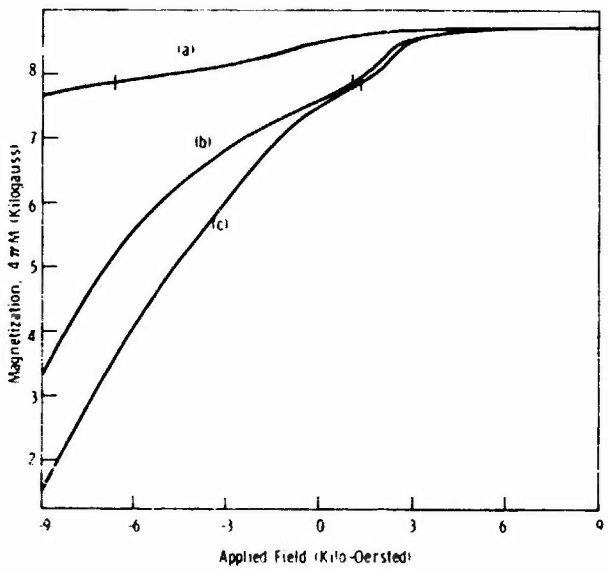

Figure 2. Magnetization Curves for the Three Cases Shown in Figure 1. The Ticks Show Values for H_k (Subtract 3740 Oersted to Obtain Internal Field). The Arrows Show the Operating Points for the Data of Figure 3.

one would have expected a reverse of the process depicted in Figure 1 and a recovery of some or all of the 180 gauss permanent loss that had been observed for this sample. (The sample had been at 215 C for about 30 hours prior to the experiment; the bulk of the permanent loss occurs within 10 hours.) In actual fact, the "heat treatments" did not lead to the recovery of any of this permanent loss. We conclude, therefore, that the mechanism of the time dependent loss is not simple diffusion. Furthermore, it is not possible to regain the virgin condition for a sample merely by holding it at the degradation temperature in a saturating field.

The most straightforward method of characterizing time dependent losses is to study their temperature dependence. For example, diffusive mechanisms yield an exponential dependence typical of an activation mechanism, whereas fluctuating fields give a linear dependence. Unfortunately,

at temperatures which give conveniently measurable effects there are clearly irreversible processes taking place. For this reason it was not possible to make a temperature-dependent study, at least not without additional information about the nature of the irreversible processes. In an attempt to gain such additional information, a series of heat treatments was carried out on sample A2. The primary goal was to reestablish a relaxation comparable to that shown in Run 1 of Figure 1, which could then be studied under different conditions of temperature and applied field. We have not so far succeeded in this attempt. A secondary goal was to observe some of the possible types of behavior. A few illustrative examples are given below. It should be emphasized that these examples are for a magnet which has been heat treated in such a way that it is not representative of production magnets. Instead, it is hoped that these runs show, to an enhanced degree, processes which also occur in high quality magnets.

In addition to the runs on the as-received sample,[1] Figure 1 illustrates two other cases. Figure 1b shows three runs measuring the time dependence of the magnetization at 215 C for sample A2 after a heat treatment of 30 minutes at 979 C. The sample was remagnetized at room temperature before each run. (The procedure is described more fully in Ref. 1.) This series represents the simplest observed behavior: the various runs display similarly shaped curves with a modest hardening from run to run. Figure 1c shows three runs on the same sample after a heat treatment of 30 minutes at 939 C. Note the unusual shape of these curves and also the softening between runs 2 and 3. Such behavior is not predicted by any of the simple theories of magnetic time effects.[4] Other samples and heat treatments gave results intermediate in complexity between those shown in Figure 1b and 1c.

The room temperature magnetization curves corresponding to the runs in Figure 1 are shown in Figure 2. The curves cover a sufficient range to define the quantity H_k, the field at which the magnetization has dropped to 90% of its remanent value. H_k is perhaps the best measure of a magnet's resistance to temperature effects.[6] The corresponding applied fields are indicated by ticks in Figure 2; internal fields may be obtained by subtracting 3740 kilo-oersted. Actually the irreversible loss was about 300 gauss for case (a); 930 gauss for case (b), run 1; 820 gauss for the other runs of (b) and 2340 gauss for case (c). It may be noted that there is a large difference in the irreversible losses between cases (b) and (c) even though there is little difference in the H_k values or in the open circuit magnetization values. On the other hand the time-dependent losses shown in Figure 1 are much larger for case (c). However, this relationship does not appear to hold in general: we have observed other cases with relatively large irreversible losses, but small time-dependent losses.

Although magnetic after-effects have been observed at room temperature in almost all permanent magnet materials, including multiphase materials based on the $SmCo_5$ structure,[5,7] the effects are very small for sintered, single-phase material.[3,7,8] For the non-optimum heat treatments of cases (b) and (c), however, we did observe very significant time effects. An example for each heat treatment is shown in Figure 3. The operating points are chosen to show effects comparable to those in Figure 1. Much greater effects were observed for treatment (c) at -9 kilo-oersted.

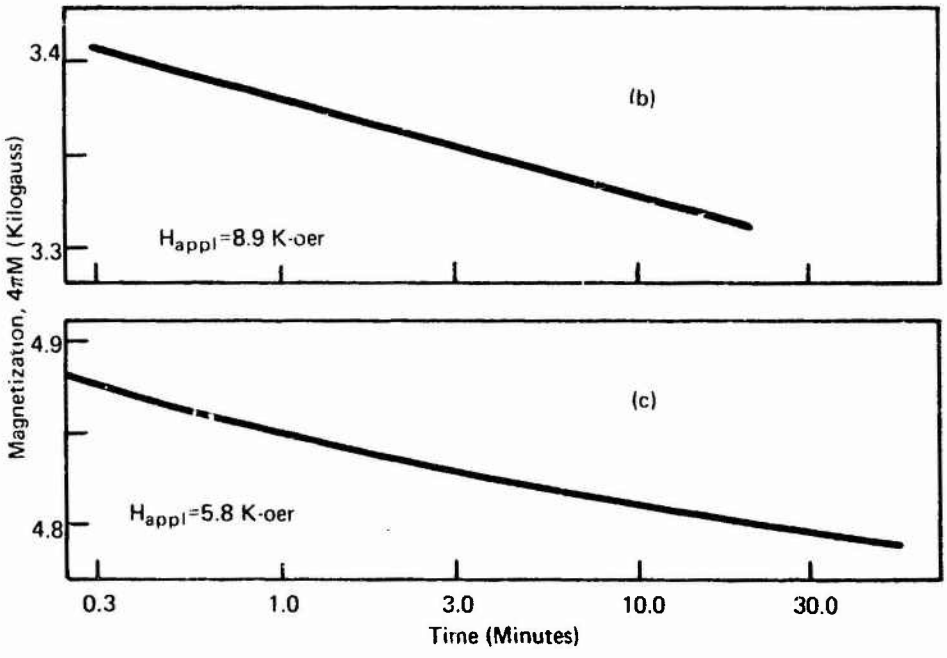

Figure 3. The Magnetic Aftereffect at Room Temperature for Two Illustrative Cases. The Effect Was Many Times Smaller for Case (a)

HIGH TEMPERATURE X-RAY STUDIES

As indicated above, we have begun a program of high temperature x-ray studies of the rare earth-cobalt magnet materials. Potentially, there is a great deal to be learned from this type of investigation. For example, heat treatment in the temperature range between 800 C and 1000 C is crucial to proper magnet production, but the actual mechanism of the changes brought about by this treatment is not understood at present. X-ray measurements made at temperature in this range would enable one to look for phase changes (or incipient phase changes), stacking faults, and perhaps other imperfections which might be responsible for the improvement in magnetic characteristics. Another property which can be studied effectively by x-rays is the thermal expansion coefficient. Directional differences in thermal expansion can be expected to be very important in determining the mechanical properties of the magnet materials through the internal stresses set up by these differences. As a concomitant to the thermal expansion, one can hope to obtain a measure of the spontaneous magnetostriction, important fundamentally, and also from a practical point of view through its effect on the internal stresses.

As a first goal in the high temperature work, we set ourselves the task of measuring the thermal expansion of $SmCo_5$ magnet material, to a point well above the Curie temperature so that magnetic effects might be

observed. This was to be done by following the angular positions of a high and a low-order Bragg reflection as a function of temperature. A scintillation counter diffractometer was set up with an iron target tube to minimize fluorescence from both cobalt and samarium. The beryllium windowed furnace, which could be set on the diffractometer, was capable of reaching over 1100 C in vacuum or over 900 C utilizing an inert gas atmosphere. Temperature measurement was by chromel-alumel thermocouple. Operation in an inert gas was considered desirable to reduce the rate of evaporation loss of samarium at higher temperatures.

The first sample studied was a piece of $SmCo_5$ magnet material oriented with the "c" axis along the diffraction vector. The initial runs to about 800 C, showed the development of strong samarium oxide lines. and the fairly rapid disappearance of the $SmCo_5$ lines above 600 C. Oxidation or at least the formation of oxide diffraction lines, was controlled by purifying the helium gas utilizing a molecular sieve at liquid nitrogen temperature, and by better outgassing techniques applied to the furnace. It was hoped that control of the oxidation in this way would result in retention of the $SmCo_5$ lines, but this proved not to be the case. Subsequent runs showed continued loss of the 1-5 lines, with the buildup of a strong reflection of "d" spacing about 2.04 A. This new peak, as discussed below, was taken to be a reflection from the close-packed planes of metallic cobalt, resulting from the loss of samarium by evaporation from the sample face. In an effort to reduce this loss, a 0.005-inch-thick sheet of Grafoil was clamped over the sample face during the runs. (Grafoil is a graphite "paper" made by Union Carbide Corp.; it was chosen for its resistance to high temperature and its low absorption for x-rays.) The results were encouraging and measurements to about 800 C seemed possible, provided they could be made fairly quickly, but the deterioration at 900 C was still excessive.

In a further effort to contain the samarium, a new sample was prepared and a 3000 Å layer of boron was evaporated onto the surface for us by Prof. R. M. Rose of M.I.T. Unfortunately, however, the retention of samarium by the boron coating was not as good as by the Grafoil. Utilizing our most successful techniques, that is, including the Grafoil shield, we attempted to measure the thermal expansion in the "c" direction, the results being given in Figure 4, which gives the shift in Bragg angle versus temperature, and in Table I, which gives the lattice parameter versus temperature. The errors result principally from the large deterioration and shift in the Bragg peak position observed before and after a run at the higher temperatures, as shown in Figure 5 and 6. Here the initially clean sample was heated to 900 C as rapidly as possible, quick measurements of the positions of the (002) and (004) peaks taken, and the sample returned to room temperature; only 12 minutes were spent above 750 C.

During our attempts to measure $SmCo_5$ at high temperature, we made some additional observations which are included here. The x-rays used in the examination of the samples penetrate only very little; $FeK\alpha$ radiation loses one half of its intensity in only seven microns. Therefore we observe only effects which occur relatively close to the surface. It is clear then that the oxide

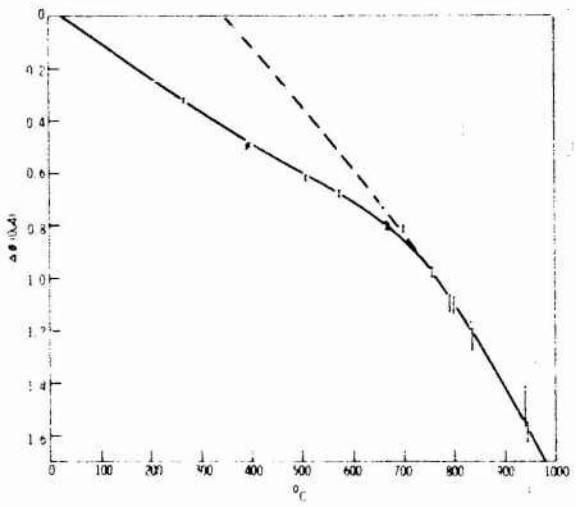

Figure 4. The Bragg Angle for the (004) Reflection of $SmCo_5$ as a Function of Temperature. The Dashed Curve Gives Schematically the Probable Form of the Curve in the Absence of Magnetic Effects. Errors Result Principally from Sample Deterioration at the Higher Temperatures.

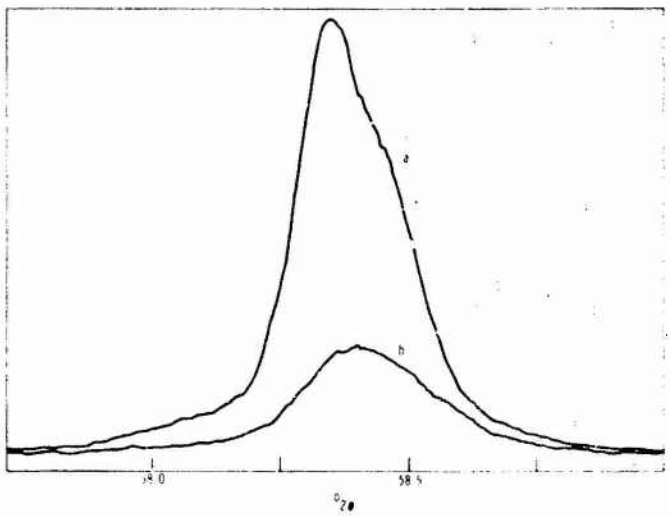

Figure 5. The (002) Reflection of $SmCo_5$ Showing (a) the Shape of the Curve Taken from a Newly Prepared Sample at Room Temperature, and (b) the Shape of the Curve, Again at Room Temperature, but After Heating to 900 C.

TABLE I. Lattice parameter "c" for $SmCo_5$ as a function of temperature. Relative errors can be obtained from the error bars in Figure 4, and range from about ± in the last place for low temperatures to ± 10 in the last place for the highest.

T (deg. C)	c (Å)
23	1.9849
100	1.9858
200	1.9869
300	1.9879
400	1.9889
500	1.9899
600	1.9908
700	1.9918
800	1.9942
900	1.9969
1000	1.9996

lines observed on early samples heated in the x-ray furnace resulted from oxygen which arrived as a contaminant in the surrounding atmosphere. This is substantiated by the fact that the oxide was readily removed from the sample by etching. The additional line which appeared after heating, referred to above as a line from metallic cobalt, was, on the other hand, very difficult to remove chemically. In an earlier experiment, however, somewhat the reverse situation was found. Here a sample of $SmCo_5$, after being sealed in quartz and heated to 1000 C, showed oxide lines and lines from a samarium-depleted phase (in this case Sm_2Co_{17}), but although the 2-17 lines were easily removed by etching, the oxide lines were not. In a companion experiment, $SmCo_5$ was sealed in quartz with samarium metal near by but not touching, and again heated to 1000 C. The magnet material showed lines of samarium-rich phases, but little or no oxidation. These observations led us to conclude that the oxygen might have its source inside the sample and arrive at the surface by diffusion. Clearly this is not the case for the majority of the oxide line intensity observed in the samples heated in the x-ray furnace, but it is possible that the samarium depletion which apparently results in the formation of the deep seated line of metallic cobalt results from internal oxidation rather than evaporation. The oxide, however, would have to be very finely dispersed to escape detection by the x-rays and we rather feel that evaporation losses are needed to explain the strong cobalt peak.

The cobalt peak itself is not well defined. It seems to appear first as a rather broad peak centered at a "d" spacing of about 2.06 Å. With further heating it grows both larger and sharper, and shifts, finally reaching, in our experiments, a "d" value of about 2.035 Å. Pearson[9] reports 2.045 to 2.054 Å for cubic cobalt and 2.035 to 2.040 Å for hexagonal cobalt, both referring to the spacing of the close-packed planes. We are unable to observe any other reflections due to the cobalt, but this may be due to an orientation relationship between the cobalt and the oriented $SmCo_5$ sample. The same line seems to appear in a heated $PrCo_5$ sample, but very much weaker.

As already mentioned, Figure 4 shows the thermal expansion data obtained from the $SmCo_5$ sample. Also shown by the dashed line is a schematic representation of the probable normal expansion curve, that is, the form of the curve to be expected in the absence of magnetic effects. One can see, for example, that on cooling from above the Curie temperature (about 720 C), the measured curve leaves the normal curve in a downward direction, indicating that the onset of magnetism causes a relative expansion in the "c" direction. (Coefficients describing the magnetostriction cannot be obtained without corresponding data for the "a" direction.)

At this time due to the problems encountered with samarium-cobalt, that effort has been temporarily abandoned, and efforts redirected toward praseodymium-cobalt. Here it is hoped that the above difficulties will be lessened, since the vapor pressure of Pr is lower than that of Sm by a factor of nearly 10 at the same temperature, and also the Curie temperature of $PrCo_5$ is lower than that of $SmCo_5$ by about 100 C. Preliminary measurements were made on a piece of cast $PrCo_5$ which, after homogenizing at 1125 C for 2-1/2 days, showed only lines of the 1-5 phase. The (222) reflection lost about 10% per 15 minutes at about 800 C, a rate which could

be tolerated during the experiments, and showed no shift in peak position on return to room temperature after heating. With this encouragement, we plan to continue the work on $PrCo_5$ in the next quarter. The first goal will be to obtain curves like Figure 4 for $PrCo_5$ in both the "a" and "c" directions.

DISCUSSION

The results described above show that not only the position of domain walls, but also the structure which pins and/or nucleates these walls, can be significantly altered at temperatures as low as 200 C, as well as at higher temperatures. Since many proposed applications involve use in the 200 to 300 C region, these structural changes must be considered. For example, a number of magnets have shown behavior exemplified by Figure 2b. In this case, it would be advantageous to remagnetize after some hours of operation at temperature. This procedure would gain even more magnetization than that lost at temperature.

The processes involved are presumably similar to those occurring at higher temperature. The x-ray studies reported here were undertaken primarily to investigate the possibility of observing these effects at temperature. Unfortunately, the experimental difficulties, particularly in the important case of $SmCo_5$, are a serious problem. Although several methods of protecting the sample were tried, none was completely successful. Thus it may be possible to make some relatively simple measurements, such as those of the lattice parameter illustrated in Figure 4, but it seems unlikely that it will be possible to make a detailed study of line shapes at temperature. These latter would be required to show, for example, the development of stacking faults.

We thank P. Weihrauch and E. Wettstein of Raytheon Company, Waltham, Massachusetts for helpful discussions.

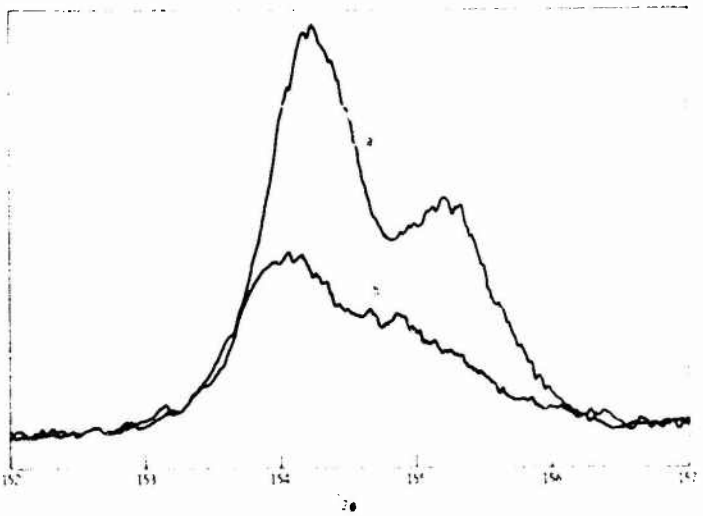

Figure 6. The (004 Reflection of $SmCo_5$ Showing (a) the Shape of the Curve Taken from a Newly Prepared Sample at Room Temperature, and (b) the Shape of the Curve, Again at Room Temperature, but After Heating to 900 C.

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